



Application Note AN-NIR-035

Quality control of polyols with NIRS

Determination of hydroxyl number according to ASTM D6342

Polyols are a broad class of organic compounds characterized by the presence of two or more hydroxyl groups. Synthetic polyols with varying chemical and physical properties are often used as raw materials in the production of polyurethanes. Quality control of these raw materials is critical prior to polymerization to avoid creating out-of-specification products. Several quality parameters can be determined in polyols like hydroxyl value, acid value (or acid number), moisture content, and viscosity. Titration is usually preferred for the analysis (ASTM D4274 for hydroxyl number, and ASTM D4662

and ASTM D7253 for acid number). This time-consuming method requires reproducible sample preparation, additional toxic and corrosive chemicals, and waste disposal, resulting in elevated running costs. Near-infrared spectroscopy (NIRS) offers a safer, faster alternative for multiparameter analysis of polyols without any sample preparation or chemical reagents. NIRS is recognized by regulatory bodies as a tool for quality control and can be used for the determination of hydroxyl values of polyols (ASTM D6342).

EXPERIMENTAL EQUIPMENT

Samples of polyols were measured in an OMNIS NIR Analyzer Liquid at 35 °C in transmission mode (1000–2250 nm). Disposable vials with a pathlength of 8 mm were used for convenience. All data acquisition and prediction model development was performed with OMNIS Software.



Figure 1. OMNIS NIR Analyzer and a sample filled in a disposable vial.

Table 1. Hardware and software equipment overview.

Equipment	Article number
OMNIS NIR Analyzer Liquid	2.1070.0010
Holder OMNIS NIR, vial, 8 mm	6.07401.070
Disposable vial, 8 mm, transmission	6.7402.240
OMNIS Stand-Alone	6.06003.010
Software license Quant Development	6.06008.002

RESULT

The NIR spectra (**Figure 2**), along with the corresponding reference values, were used to create prediction models for quantification of viscosity, hydroxyl number, acid value, and moisture in polyol samples.

The quality of the prediction models was evaluated

using correlation diagrams (**Figures 3–6**). These diagrams display very high correlations between the NIR predictions and the measured values of different parameters using wet chemical methods. The respective figures of merit (FOM) display the expected precision during routine analysis.

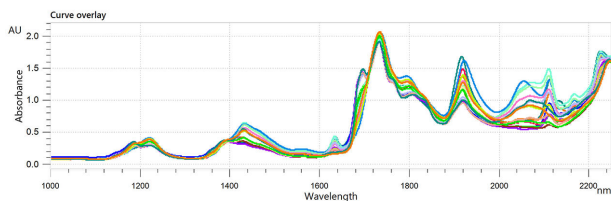


Figure 2. Stacked collection of NIR spectra from polyol samples analyzed with the OMNIS NIR Analyzer Liquid.

RESULT HYDROXYL NUMBER IN POLYOLS

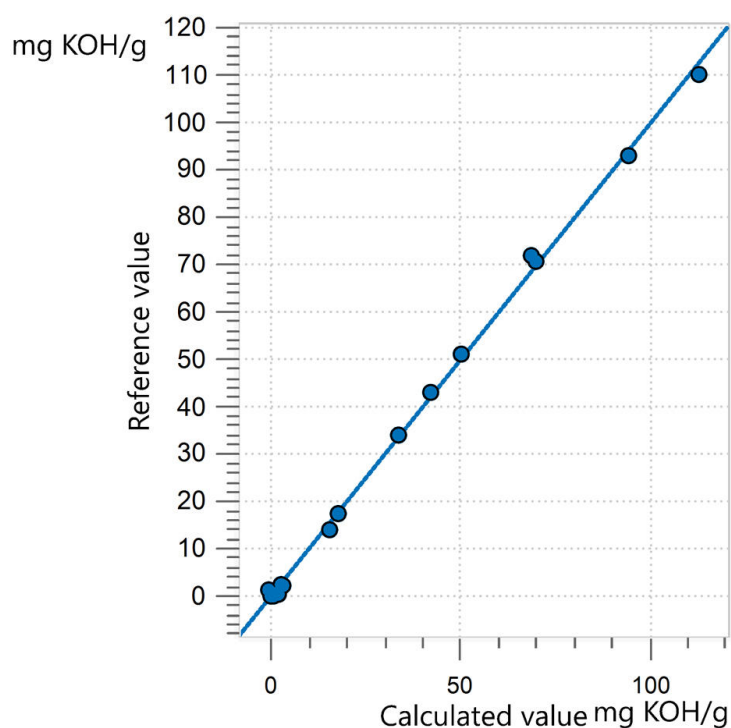


Figure 3. Correlation diagram and the respective figures of merit for the prediction of the hydroxyl number in polyols using an OMNIS NIR Analyzer Liquid. The hydroxyl number lab values were evaluated using titration.

R^2	SEC (mg KOH/g)	SECV (mg KOH/g)
0.998	1.17	1.42

RESULT ACID NUMBER IN POLYOLS

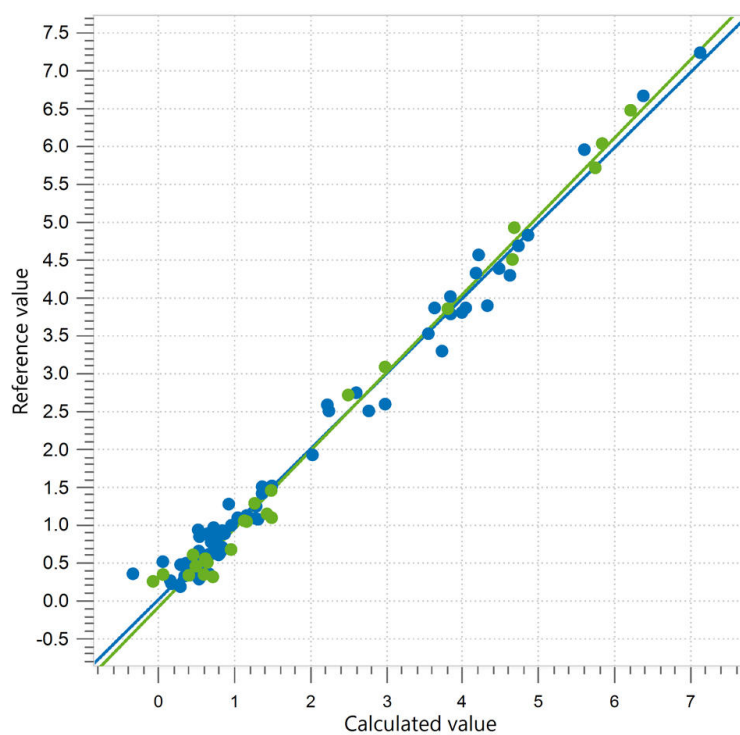


Figure 4. Correlation diagram and the respective figures of merit for the prediction of polyol acid number using an OMNIS NIR Analyzer Liquid. Titration was used to determine the laboratory acid number values.

R^2	SEC (mg KOH/g)	SECV (mg KOH/g)	SEP (mg KOH/g)
0.990	0.13	0.20	0.21

RESULT MOISTURE CONTENT IN POLYOLS

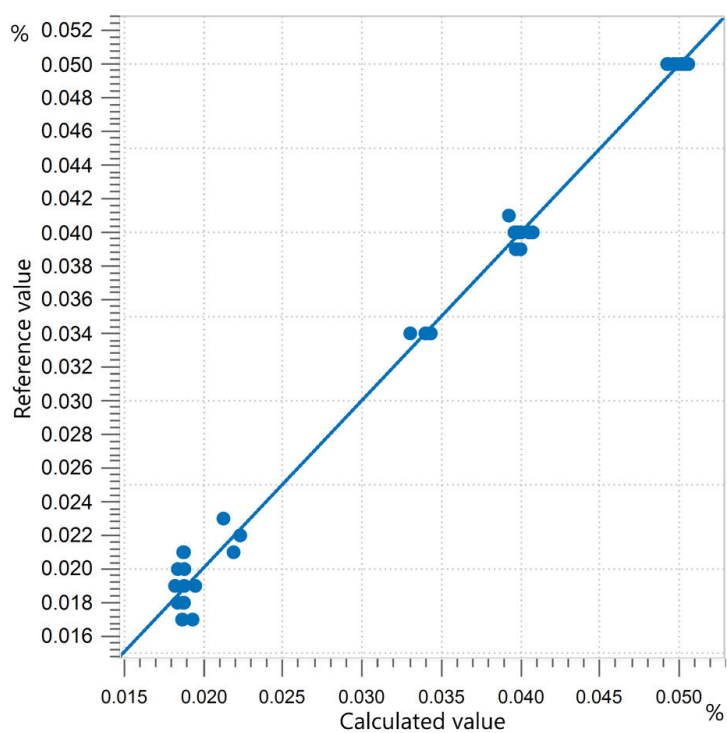


Figure 5. Correlation diagram and the respective figures of merit for the prediction of polyol water content using an OMNIS NIR Analyzer Liquid. Karl Fischer titration was used to determine the laboratory values for moisture content in polyols.

R^2	SEC (ppm)	SECV (ppm)
0.994	9	10

RESULT VISCOSITY IN POLYOLS

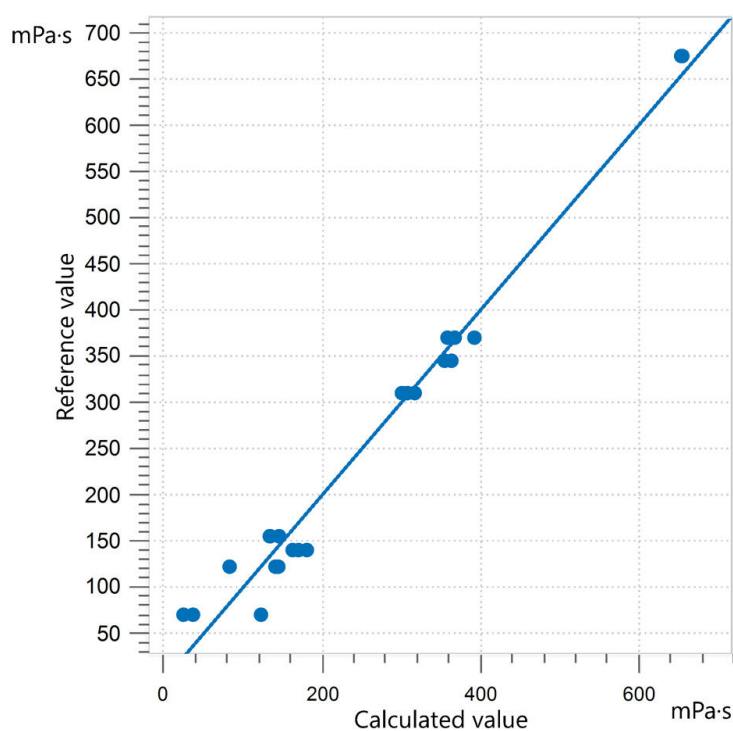


Figure 6. Correlation diagram and the respective figures of merit for the prediction of viscosity in polyols using an OMNIS NIR Analyzer Liquid. The viscosity lab values were evaluated with a viscometer.

R ²	SEC (mPa·s)	SECV (mPa·s)
0.978	20.25	25.59

CONCLUSION

This Application Note shows the feasibility of using NIR spectroscopy for the analysis of hydroxyl number, moisture, acid number, and viscosity in polyols.

Compared to wet chemical methods, running costs are significantly lower when using NIR spectroscopy (Figure 7).

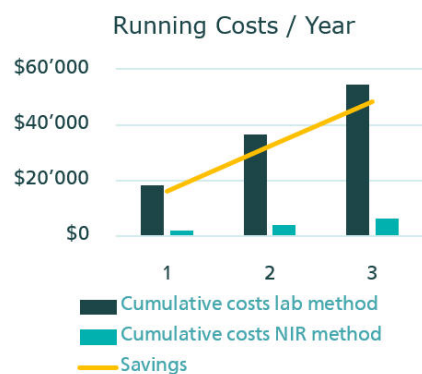


Figure 7. Comparison of running costs for the determination of the hydroxyl number with a standardized method (titration) and NIR spectroscopy.

	Lab method	NIR method
Number of analyses (per day)	10	10
Cost of operator (per hour)	\$25	\$25
Costs of consumables and chemicals OH-number	\$6	\$1
Time spent per analysis	5 min	1 min
Total running costs (per year)	\$18,188	\$2,063

CONTACT

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CONFIGURATION



OMNIS NIR Analyzer Liquid

Nahinfrarot-Spektrometer für Flüssigproben.

Der OMNIS NIR Analyzer ist die nach Schweizer Qualitätsstandards entwickelte und produzierte Nahinfrarot-Spektroskopie (NIRS) Lösung für die Routineanalytik entlang der gesamten Produktionskette. Die Nutzung neuester Technologien und die Einbindung in die moderne OMNIS Software spiegeln sich in der Geschwindigkeit, der Bedienbarkeit und dem flexiblen Einsatz dieser NIR-Spektrometer wider.

Die Vorteile des OMNIS NIR Analyzer Liquid im Überblick:

- Messungen von Flüssigproben in weniger als 10 Sekunden
- Temperaturkontrolle an der Probe von 25°C – 80°C
- Automatische Erkennung des Einsetzen und der Entnahme des Probengefäßes
- Einfache Einbindung in ein Automationssystem oder Verknüpfung mit weiteren Analysetechnologien (Titration)
- Unterstützung zahlreicher Probengefäße mit unterschiedlicher Pfadlänge



Halter OMNIS NIR, Vial, 8 mm

Vialhalter für den OMNIS NIR Analyzer für 8 mm Einwegvials (6.7402.240).



Einwegvial, 8 mm, Transmission, Anz. 100

100 Einwegvials aus Glas (Borosilikat) mit einer optischen Pfadlänge von 8 mm für Analysen von Flüssigkeiten in Transmission. Die Einwegvials werden mit den zugehörigen Verschlussstopfen (Stückzahl = 100) geliefert.

Kompatibel mit:

- Halter OMNIS NIR, Vial, 8 mm (6.07401.070)
- DS2500 Halter für 8 mm Einwegvials (6.7492.020)

OMNIS
A WHOLE NEW LEVEL OF PERFORMANCE

OMNIS Stand-Alone Lizenz

Ermöglicht den Stand-Alone Betrieb der OMNIS Software auf einem Windows™ Computer.

Merkmale:

- Die Lizenz enthält bereits eine OMNIS Geräte Lizenz.
- Muss über das Metrohm Lizenzierungsportal aktiviert werden.

OMNIS
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Softwarelizenz Quant Development

Softwarelizenz für die Erstellung und Bearbeitung von Quantifizierungsmodellen in einer Stand-Alone OMNIS Software Installation.